

EFFECTIVE DATE

December 10, 1996

LANL-CST-DP-103, R1

Page 1 of 10

IDENTIFICATION, STORAGE, AND HANDLING OF SAMPLES FOR THE WATER MOVEMENT TEST

LOS ALAMOS QUALITY PROGRAM



APPROVAL FOR RELEASE

J. T. FABRYKA-MARTIN - PREPARER

Signature on file

DATE

J. T. FABRYKA-MARTIN - PRINCIPAL INVESTIGATOR

Signature on file

DATE

M. J. CLEVINGER - QUALITY ASSURANCE PROJECT LEADER

Signature on file

DATE

Los Alamos
Yucca Mountain Site
Characterization Project

HISTORY OF REVISION

REVISION NO.	EFFECTIVE DATE	PAGES REVISED	REASON FOR CHANGE
R0	12/21/94	N/A	Supersedes LANL-INC-DP-87 to reflect that samples for this activity are stored and handled at LANL facilities.
R1	12/10/96	All	Revised to comply with LANL-YMP-QP-06.3 requirements.

Los Alamos

Yucca Mountain Site
Characterization Project

IDENTIFICATION, STORAGE AND HANDLING OF SAMPLES FOR THE WATER MOVEMENT TEST

1.0 PURPOSE

This detailed technical procedure (DP) describes procedures used to track the receipt, storage, history and disposition of samples at LANL facilities for the Yucca Mountain Site Characterization Project (YMP). Samples and their derivatives used in YMP studies and analyses require chain-of-custody documentation and appropriate handling procedures and storage facilities to ensure their integrity and traceability.

2.0 SCOPE

This DP applies to LANL and LANL-subcontractor YMP personnel who receive or use geologic samples or their derivatives in YMP investigations as part of the Water Movement Test task for Los Alamos National Laboratory (LANL). This DP does not control tests, operations, or processes conducted on samples, nor does it control the generation and treatment of data. These aspects of sample treatment are governed by other DPs.

3.0 REFERENCES

LANL-YMP-QP-02.7, Personnel Training
LANL-YMP-QP-03.5, Documenting Scientific Investigations
LANL-YMP-QP-08.1, Identification and Control of Samples
LANL-YMP-QP-17.6, Records Management
LANL-YMP-DP-89, Procedure for Sieving Soil and Rock Samples
LANL-CST-DP-90, Measurement of Moisture Content of Soil Samples
LANL-CST-DP-93, Step-Leaching Procedure for Extracting Soluble Chloride and Bromide
LANL-CST-DP-97, Preparation of Carrier Solution for Chlorine-36 Samples
LANL-CST-DP-108, Collection of Field Samples for the Water Movement Test
LANL-CST-DP-109, Particle Size Reduction of Samples

4.0 DEFINITIONS

4.1 Sample

A sample is a part of a population whose properties are studied to gain information about the whole or group. Examples of samples include surface and subsurface soils, rocks and fluids, drill cores, cuttings, muck from underground excavations, and biota. For Los Alamos purposes, sample is synonymous with specimen. In this DP, the term sample is used to refer to parent, split and subsplit materials (see below).

4.2 Parent Sample

A parent sample is that mass of sample which is received at LANL, and which originated from the field, from the Sample Management Facility (SMF), from other investigators, or from commercial or other sources, with the intent of use for site characterization under YMP auspices. Each parent sample is assigned a unique LANL number which is traceable to a unique SMF designation. The LANL number is retained for any residue remaining after splits are made. Other markings may also be used unofficially; such markings may be desirable in order to tag the sample as coming from a specific field site or location in a drill core, or for the convenience of quick recognition.

4.3 Split

Multiple splits or aliquots may be extracted from a parent sample to allow for various types of analyses, for studying compositional variability in a heterogeneous sample, for sharing of sample portions with other researchers, or for allowing replicate analyses of a homogeneous sample. Each split is assigned the unique LANL number of the parent material plus a unique split designation (for the LANL number). For convenience and to allow for quick recognition, other unofficial markings and shorthand notations may also be used to label containers holding samples while processing them according to a specific DP or notebook procedure.

Examples of splits are chips, powder prepared from fragments, aliquots of otherwise homogeneous material, and mineral separates (e.g., fracture fill minerals isolated from a hand specimen or drillcore) such as those isolated from the bulk rock following DP-109. Incidental fragments (e.g., when a piece breaks off from the parent) are stored with the parent and are not considered splits.

4.4 Subsplit

Subsplits are specimens extracted from a split, with each subsplit being uniquely identified as discussed in section 6.5.3. Examples of subsplits are sieved fractions prepared from a split according to DP-89 or aliquots prepared during the step-leaching procedure according to DP-93.

4.5 Chain of Custody

Chain-of-custody documentation is written documentation showing the transfer of a sample from its point or origin to its usage in a given laboratory study or analysis and, if appropriate, its return to storage or to other personnel.

4.6 Sample Inventory Logbook

The Sample Inventory Logbook consists of one or more notebooks and any associated binders that are dedicated to recording or logging the receipt, origin, location of storage, analytical results (if appropriate), current custodian (if the samples are not in a LANL storage area), and final disposition of samples used at LANL for the Water Movement Test task. Maintenance of the logbook follows guidelines in QP-03.5.

5.0 RESPONSIBILITIES

The following personnel are responsible for the activities identified in Section 6.0 of this procedure.

- Principal Investigator (PI) for the Water Movement Test
- YMP personnel performing work to the procedure

6.0 PROCEDURE

The use of this procedure must be controlled as follows:

- If this procedure cannot be implemented as written, YMP personnel should notify appropriate supervision. If it is determined that a portion of the work cannot be accomplished as described in this QP, or would result in an undesirable situation, that portion of the work will be stopped and not resumed until this procedure is modified, replaced by a new document, or until current work practice is documented in accordance with QP-03.5, Section 6.1.6.
- Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used.
- When this procedure becomes obsolete or superseded, it must be destroyed or marked “superseded” to ensure that this document is not used to perform work.

6.1 Principle

Samples used in YMP studies and analyses for site-characterization require chain-of-custody documentation and appropriate handling procedures and storage facilities to ensure their integrity and traceability.

6.2 Equipment and Hardware/Software

6.2.1 Equipment Malfunctions

No equipment is required to be used in the performance of this DP.

6.2.2 Safety Considerations

Good laboratory and scientific practices are used to protect against injury. Applicable LANL and/or LANL-contractor safety practices for conducting work will be followed, as appropriate.

6.2.3 Special Handling

Care must be taken to minimize any potential for contamination of the sample with the analyte of interest (usually chloride or ^{36}Cl).

6.3 Preparatory Verification

6.3.1 Hold Points

The Sample Inventory Logbook, described in section 6.4, is used to record receipt of samples into the LANL storage areas, taking of samples from these storage areas, and return of samples to the storage areas after use. LANL numbers are assigned to new parent samples as described in section 6.5. Before any LANL number is assigned, the person assigning the number checks the last number in the logbook so as to prevent duplication of a number. For the same reason, when sample splits are subsequently made, the person assigning the split number checks the logbook before assigning a split identification. The purpose of these verifications is to prevent repetition of sample identifiers that would lead to ambiguities in sample history.

6.3.2 Calibration - N/A

6.3.3 Environmental Conditions

Special temperature or humidity controls are not required for most samples used in the Water Movement Test study, except for soil samples collected for moisture content determinations. As described in DP-108, these samples must be stored in airtight containers and in a cool location until their initial (wet) weights have been obtained following the procedure in DP-90; subsequently, no special environmental conditions are required. For samples intended for chloride, bromide, or chlorine isotopic analyses, sealed containers are required to minimize the potential for cross-contamination. Any other special environmental conditions required for sample storage are provided by the responsible PI, noted in the Sample Inventory Logbook (see section 6.4), and marked on the outside of the sample container. The location where such special samples are kept is recorded in the Sample Inventory Logbook.

Sample security is important. The sample storage area is kept locked during nonworking hours.

6.4 Control of Samples

6.4.1 Logbook Entries

Entries in the Sample Inventory Logbook conform with the requirements of the procedure for scientific investigations (QP-03.5). A section of the logbook is reserved for a table of contents or index that indicates the page number(s) on which the records for a particular sample type may be found.

On the sample page, the initial entry indicates the original of the sample, date of receipt, approximate quantity and form in which it was received, name of the sender, a unique identifier assigned by the user (see section 6.5), and other entries as listed in Attachment 1. The sample quantity may be specified either by volume or by mass and may be obtained by a visual estimate or from the shipping documents; a calibrated balance is not required for the purposes of the logbook.

Subsequent entries represent a chronology of the history of the material. Required entries are listed in Attachment 1.

6.4.2 Logbook Binder

Separate loose-leaf binder(s) are considered part of the Sample Inventory Logbook and are maintained and used in accordance with the requirements for scientific investigations (QP-03.5). These binders contain records of forms and correspondence generated during the acquisition and transfer of samples as well as separate copies of analytical results and/or other tests performed on soil, rock, water or other samples if such results are not already included in another logbook.

6.5 Implementing Procedure

6.5.1 Inspection of Samples Upon Receipt

6.5.1.1 The user physically inspects any sample received (e.g., from the field, the Sample Management Facility, or another investigator) in order to determine whether it may be unacceptable for the purpose for which it was intended. In cases where a sample has been damaged or deteriorated in transit, the user contacts the PI to discuss sample disposition.

6.5.1.2 The user verifies the identification numbers or letters found on the sample or its container against accompanying documents or with previously received documentation. Samples received from the YMP Sample Management Facility must have a unique bar code or other type of identifying label accompanying the sample and/or its container.

- 6.5.1.3 If the required labels or barcodes are missing or do not match the documentation, the user contacts the PI to discuss sample disposition.
- 6.5.1.4 The user files shipping documents in the Sample Inventory Logbook (e.g., in a loose-leaf binder considered to be part of the logbook).

6.5.2 Assignment of LANL Identifier to Parent Sample

The user assigns a unique alphanumeric identifier to each parent sample upon receipt. Any format can be used provided it is logical, systematic and described in the Sample Inventory Logbook. Most commonly, identifiers for the Water Movement Test consist of one or two letters indicating the type of sample, followed by a three-digit number in chronologic sequence for a particular type of sample. Examples of letters used to indicate the sample type are: SM for soil moisture samples; ST for soil trench or profile samples; R for rock cutting samples; E for hand specimens of rock from the Exploratory Studies Facility; W for water samples; DC for drillcore samples; and C for carrier salt, as defined and used in DP-97.

6.5.3 Assignment of LANL Identifiers to Splits and Subsplits

- 6.5.3.1 The user assigns a unique identifier to each portion removed from a parent sample. This identifier is derived from that of the parent sample, so as to make it easy to track the history of any given sample. Any format can be used provided it is logical, systematic and described in the Sample Inventory Logbook. Most commonly, split identifiers for the Water Movement Test consist of the parent identifier followed by a hyphen, then a number corresponding in sequence in formation of splits from that sample. Identifiers for subsplits generally consist of the split identifier to which is appended a letter of the alphabet corresponding in sequence in formation of subsplits from that split.

NOTE: For example, the first three splits of sample R142 are assigned the identifiers R142-1, R142-2, and R142-3. The first three subsplits of R142-2 are assigned identifiers R142-2A, R142-2B, and R142-2C.

- 6.5.3.2 The user does not need to assign a split identifier if the sample can be safely assumed to be homogeneous and stable with respect to the feature of interest (e.g., chloride in a water sample) or if the entire parent sample is used for a particular analysis (e.g., moisture analysis).

- 6.5.3.3 The user enters into the Sample Inventory Logbook or into the laboratory notebook a brief description of the procedure used to create the split or subsplit. Split identifiers are recorded in the Sample Inventory Logbook. Recording of subsplit identifiers in the logbook is not required.

6.5.4 Assignment of LANL Identifiers to Derivation Samples

Although a sample may be transformed by the procedure done to it, it remains the original identifier assigned to it, or some derivative thereof as described in section 6.5.3. However, for convenience in tracking the chlorine isotopic results, the user may assign a new unique alphanumeric identifier to each silver chloride precipitate prepared from a given sample. Correspondence between the original sample identifier and that for the silver chloride precipitate is then recorded in a logbook in a manner that makes it easy to track sample results.

6.5.5 Transfer of Sample Into or Out of Storage

- 6.5.5.1 Upon receipt of a sample, the user may transfer it directly to the appropriate laboratory for use or else store it in a LANL sample storage area.
- 6.5.5.2 Each time a sample is removed from storage for use, the user notes its transfer in the logbook, along with other information listed in Attachment 1, as appropriate.
- 6.5.5.3 The user makes an entry in the sample logbook when the parent material is used up, discarded, returned to the SMF for archiving, or otherwise removed from the LANL inventory.

6.6 Data Acquisition and Reduction

The only method of data acquisition governed by this procedure is an entry in a logbook. Information to be recorded is described in section 6.5. No data analysis is involved in this DP. Documentation in notebooks is acceptable if entries meet relevant requirements of QP-03.5.

6.7 Potential Sources of Error and Uncertainty

Potential sources of uncertainty concerning sample origins or history may arise if (a) two different samples (whether parent, split, or subsplit) are assigned the same identifiers and cannot be distinguished by other markings or by recorded descriptions, (b) sample splits or subsplits are assigned incorrect identifiers, or (c) the sample identifier is rubbed off, washed off, or otherwise missing from the container.

- 6.7.1 To minimize the possibility of assigning incorrect identifiers to sample splits and subsplits, the user enters a brief description of the procedure used to produce the split or subsplit, and/or the procedure to be performed on it, in the Sample Inventory Logbook at the time when the sample is first assigned its split or subsplit identifier.
- 6.7.2 The user investigates any duplication of identifiers to determine whether the split or subsplit features are sufficient to distinguish between the materials with duplicated identification. If so, then the user documents the basis for distinction in the Sample Inventory Logbook or in the laboratory notebook, as appropriate, and assigns appropriate unique identifiers to the materials involved.
- 6.7.3 If a unique sample identity cannot be established for a given sample, then the user labels or marks any data obtained for that sample during the period of uncertainty so that it is clear those data cannot be used for quality-affecting purposes.
- 6.7.4 Irrecoverable loss of adequate identifying marks from any parent or split, or illegibility of such marks to the extent that they cannot be read or reconstructed unambiguously, require that the user either discard the parent or split material, or else clearly label it as not usable for producing QA-traceable data (although it may still be used for scoping activities).

7.0 RECORDS

Records generated as a result of the proper execution of this DP are entries in the Sample Inventory Logbook, which includes any associated binder(s). These entries are listed in Attachment 1. These records are generated in accordance with the procedure QP-03.5 and are submitted to the Records Processing Center as stand-alone records when closed out, in accordance with QP-17.6.

8.0 ACCEPTANCE CRITERIA

The criteria that show that this procedure has been correctly implemented are the records identified in Section 7.0. All parent, split and subsplit materials are acceptable for use unless sample traceability is lost or unless sample numbers become duplicated and sample origins cannot be uniquely reconstructed, in which case the procedures in section 6.7 and QP-08.1 are followed.

9.0 TRAINING

This DP requires “read-only” training. Training of personnel to this DP is documented pursuant to QP-02.7.

10.0 ATTACHMENTS

Attachment 1: Checklist of required entries in the Sample Inventory Logbook (1 page)

CHECKLIST OF REQUIRED ENTRIES IN THE SAMPLE INVENTORY NOTEBOOK

In addition to the initial notebook entries required by QP-03.5, the following initial entries are required for each sample as it is received:

- Date of receipt
- Origin of sample (e.g., borehole identifier)
- Approximate quantity (either volume or mass)
- Form in which received (e.g., soil, water, cuttings)
- Name of sender or originator
- Unique identifiers and relevant label information found on sample or its container (e.g., field ID and/or SMF barcode)
- Date and initials of person verifying sample identity
- Unique LANL identifier assigned following this DP
- Location of storage
- List of accompanying documents filed in logbook binder (see below)
- Special environment conditions required for sample storage (if any)

The following subsequent entries are required if a sample is withdrawn or returned to storage, depleted in an experiment, or discarded, as appropriate:

- Name of person making withdrawal
- Date of withdrawal
- Purpose of withdrawal (brief description of planned sample treatment, experiment or analysis)
- Unique split identifier assigned per this DP (if required)
- Notebook identifier in which subsequent sample history can be found (if applicable)
- Approximate quantity (either volume or mass) withdrawn
- Comment when parent sample is used up, discarded, or otherwise removed from the LANL inventory

The following entries are required whenever appropriate:

- Changes (if any) from the methods described in this DP
- Problems (if any) which may pose a potential source of error or uncertainty for results
- Basis of distinction (if any) between samples with duplicated identification
- Definitions of any letter codes used for LANL identifiers that are not defined in this DP

The following are examples of the types of documents which are included in the logbook binder; as appropriate (if not included in other YMP record packages, notebooks or logbooks):

- Shipping documents
- Correspondence received concerning samples
- Sample transfer of custody receipts
- Analytical results received from subcontractors